Activated Carbon preparation from Groundnut Cake and its application for Congo Red dye removal

Thesis Submitted

by

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CERTIFICATE

This is to certify that the thesis entitled **"Activated Carbon preparation from Groundnut cake and its use in Adsorption studies of Congo Red dye"** submitted to the National Institute of Technology, Rourkela by **ANINDITA DEY**, Roll No. **214CH1097** in partial fulfillment of the requirements for the award of the degree of **Master of Technology** in **Chemical Engineering**, is an authentic and a bonafide research work carried out by her under my supervision and guidance. The thesis, which is based on candidate's own work, has not been submitted elsewhere for any degree/diploma.

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ABSTRACT

The preparation of activated carbon by the activation of groundnut cake using sulphuric acid and its ability to remove the Congo Red (CR) dye from the aqueous solution in batch process were presented in this study. The effect of contact time, adsorbent dose, initial pH of the dye solution, temperature and initial dye concentration on the removal of CR dye were studied. The activated carbon was characterised by Iodine no., Scanning electron microscopy (SEM) and Fourier transform infra-red (FTIR) spectroscopy. Results showed that the activated carbon prepared has a good no. of active sites and pores for adsorption. Adsorption studies showed that adsorption was more effective in the acidic medium and increase with contact time, initial dye concentration adsorbent dosage and temperature. The adsorption capacity of the prepared groundnut cake activated carbon was 3.2 mg/g. The kinetic study of adsorption was better described by Pseudo-2ndorder kinetic model and Freundlich isotherm describes the CR adsorption equilibrium data better than Langmuir isotherm.

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NOMENCLATURE:

A = % ash content in the sample

M = % moisture content in the sample

R = Universal gas constant (8.314 J/mol K)

T = Absolute temperature (K)

V = Volume of the solution taken in flask (l)

W = Amount of adsorbent (g)

C_0 = Initial dye concentration (mg/l)

C_e = Final dye concentration (mg/l)

K_1 = Pseudo-first-order rate constant

K_2 = Pseudo-second order rate constant

K_f = Freundlich constant (l/mg)

K_{id} = Inter particle diffusion rate

V_m = % volatile matter in the sample

W_1 = Weight of the empty crucible (g)

W_2 = Weight of the crucible with sample before put in to the furnace (g)

W_3 = Weight of the crucible with sample after taking out from furnace (g)

b = Langmuir constant

t = Time (min.)

q_t = Amount of solute adsorbed per unit weight of adsorbent at any time t (mg/g)

q_e = Amount of solute adsorbed per unit weight of adsorbent at equilibrium (mg/g)

q_m = Monolayer adsorption capacity of the adsorbent (mg/g)

Greek letters:

α = Initial adsorption rate (mg/g. min)

β = desorption constant (g/mg)

Chapter 1
Introduction

1. INTRODUCTION

Water pollution is a major global problem of present time. One of the main constituent of the water pollution is the effluent from various industries which contain various pollutants typically dyes. The presence of dye or their degraded products even in very low concentration in water can cause serious human health disorder like hemorrhage, ulceration of skin can cause severe damage to kidneys, liver, brain reproductive system and central nervous system.Hence it is necessary to findeconomical and efficient methods for successively remove them.

There are several techniques for removal of dyes and decolorization, (1) Physical methods, such as adsorption on peat, activated carbon, fly ash, wood, silica and other process like ion-exchange, membrane filtration, coagulation and reverse osmosis; (2) Chemical methods, involves oxidation using oxidizing agents like Fenton's reagent , ozone, sodium hypochlorite etc. Others include photochemical methods and electrochemical degradation; (3) Biological methods, using fungi and bacteria as the dye degrading agent. Some of these techniques are effective to remove the dye from solution. However these processes have limitations such as more uses of chemical, accumulation of concentrated used sludge that has disposal problem, lack of effective color reduction and expensive.

Congo Red (CR) dye is particularly used in various industries such as textile, paper and acrylic etc. It is highly unsafe if present in the effluent stream without proper treatment. Techniques employed for removal of CR dye include photo-catalytic degradation, photo-degradation, adsorption andbioremediation. Although removal of dyes from aqueoussolution through adsorption by activated carbon is quite effective as compare to other techniques, butcommercial application of activated carbons is still restricted due to its regeneration and production costs.

Adsorption is the accumulation of adsorbate on to the surface of solids adsorbent. Adsorption is a process where particles from solution are bound to the surface of adsorbent by physical or chemical forces. This technique is better than other color removal methods in term of low introductory expense, straightforwardness of configuration, simplicity of operation, and non-toxicity of the used adsorbent contrasted with other waste water treatment techniques.The availability cost, effectiveness and adsorption capacity are the main criteria for the activated carbon to utilize them as adsorbent to remove the dyes from waste water. Activated carbon is

used as an absorbent in many industries for removal of dyes from wastewater. Activated carbon has good adsorption capacity because of its micro-porous, pores structure with high surface area and it also shows the stability in acidic and basic medium. However in present time because of high cost of commercial grade activated carbon, activated carbon is preparing from cheaper precursors, such as hen feather (Mittal, 2006) and rise husk (Sharma et al. 2009). Various research studies had shown that many lesser price precursors are used as an adsorbent for dye removal from aqueous solutions by adsorption. However, very few low price activated carbons can be well used to remove dye from the waste water.

Nowadays, there is a more interest in finding less expensive and effective alternatives to the existing commercial activated carbon. Exploring cheap and effective activated carbon may contribute to environment sustainability and give benefits for future industrial applications. Objective of the present study is to use groundnut cake for preparation of activated carbon. It also includes study of the capacity of activated carbon for removal of CR from aqueous solution as there is limited research had done on application of the groundnut cake as a precursor for activated carbon. The prepared activated carbon was characterized using Iodine value test, Sem analysis and FTIR analysis. Effect of contact time, initial dye solution concentration, temperature, initial solution pH, activated carbon dose on the adsorption of the CR dye onto the prepared activated carbon were examined. The pseudo-1st order, pseudo-2nd order, Elovich equation, intra-particle diffusion models are used to correlate the adsorption kinetic data for adsorption of CR on the prepared activated carbon. Freundlich and Langmuir isotherms were were used to fit the equilibrium data for adsorption of CR on activated carbon.

Chapter 2
Literature review

2. LITERATURE REVIEW

2.1 Activated carbon

Activated carbon (AC) is a non-graphitic, non-graphitizable carbon with a highly disordered microstructure. It is well known for high adsorption capacity due to its high surface area and porosity. Activated carbons are carbonaceous materials that can be distinguished from elemental carbon by the oxidation of the carbon atoms found on the outer and inner surfaces. These materials are characterized by their extraordinary large specific surface areas, well-developed porosity and tunable surface-containing functional group. For these reasons, activated carbons are widely used as adsorbents for the removal of organic chemicals and metal ions of environmental or economic concern from air, gases, potable water and wastewater. The surface oxygen functional groups can be easily introduced to the carbon by different activation methods including dry and wet oxidizing agents. Dry oxidation methods involve the reaction with hot oxidizing gas such as steam and CO_2 at temperatures above 700^{o} C. Wet oxidation methods involve the reaction between the carbon surface and solutions of oxidizing agents such as phosphoric acid H_3PO_4, nitric acid HNO_3, hydrogen peroxide H_2O_2, zinc chloride $ZnCl_2$, potassium permanganate $KMnO_4$, ammonium persulphate $(NH_4)_2SO_8$, potassium hydroxide KOH, etc.

Activated carbon usually increases the cost of the treatment process. Its economical drawback has stimulated the interest to utilize cheaper raw materials for the production of activated carbon. Consequently, a wide variety of agricultural by-products and wastes has been investigated as cellulosic precursors for the production of activated carbon in addition to hard wood and bituminous coal. These precursors include coconut shell and wood, Olive stones , sugarcane bagasse, pecan shells, palm seed, apple pulp, rubber seeds and molasses. Commercial activated carbons are commonly produced from naturally occurring carbonaceous materials such as coal, wood and peat. Due to the growing need for activated carbons in our society and the high cost of raw materials and production, many researchers have attempted various wastes such as tires, resins, agricultural byproducts; and dried sewage sludge as raw materials and proposed new production methods for activated carbons with potential applications in pollution control. Furthermore, more interest has been devoted to utilize some wastes of carbonaceous materials such as paper mill slude, old newspapersand waste tires. Recently, activated sludge has been

produced as a result of wastewater treatment activities and has emerged as an interesting option for the production of activated carbon.

Production of AC is achieved typically through two routes, physical activation and chemical activation.

Kang et al.,(2006) prepared activated carbon from paper mill sludge using potassium hydroxide as activating agent. Based on the experimental observations, it was identified that the temperature and impregnation ratio of activating agents have the greatest influence on the quality of the activated carbon during the activation step. Increase in reaction time also resulted in better activation. However, a higher temperature (800-900^{o}C) resulted in severe activation that had a detrimental impact on the development of porosity on activated carbons, and for higher impregnation ratio (above 2.0), decreases of adsorptive capacity were observed, which can be attributed to the severe release of tar resulting from severe activation of char, and resulting in a larger widening of porosity. Longer reaction times, also, resulted in decreased adsorptivity, which was due to coalescence or widening of previously formed pores. Activated carbon prepared from activation at 700^{o} C for 60 min by using potassium hydroxide-to-char of 1.0 was found to have maximum iodine number.

Ahmad et al.,(2007) prepared activated carbon from coconut husk using potassium hydroxide as activating agent. The optimum conditions for preparing activated carbon from coconut husk for adsorption of 2,4,6-TCP were found to be activation temperature of 750^{o}C, activation time of 2.29 h and KOH:char impregnation ratio of 2.91.

Carvalho et al.,(2008) prepared activated carbon from cork powder using potassium hydroxide as the activating agent. From the study of the ratio between the activating agent and the cork powder, as well as from the changes in the activation temperature, it was concluded that when using equal weight amounts of KOH and cork powder, and for an activation temperature of 800 °C, the activated carbon obtained presents values of specific surface areas and microporous volumes that can reach 1300 m^2 /g and 0.56 cm^3 /g, respectively.

2.2 TEXTILE ORGANIC DYE

A dye is a colored substance that has an affinity toward the substrate to which it is being applied. Dye can be prepared by two types either by natural process and other by synthetic process. The natural dyes were used in textile industries until 1866, and these dyes are based on the extracts

from the vegetables and animals. It is known that indigo dye was extracted from the indigo plant since 3000 BC in India.

Dye is applied in an aqueous solution that needs a mordant component to increase the easiness and fastness of the dye to color the substrate. A dye loses its color when some its chemical properties changes. Dyes containauxochromes which are called color helpers. Carboxylic acid, sulfonic acid, aminogroups are some common of the auxochromes. These auxochromeschanges the solubility of the dye in aqueous solution. Due to presence of these chemicals in the dye it is of more concern to remove the dye from the effluent of the industries.

The World Bank date shows that almost 15-20% of global industrial water pollution is due to the treatment and dyeing of textiles. Nearly 10-15% of dyes are released through effluent during dyeing process which makes the effluent highly colored.

2.3 ADSORPTION

Adsorption occurs when a solute accumulates on the surface adsorbent to form a molecular film ofadsorbate. Adsorption process is widely used industrial applications process as in water purification and synthetic resins. There are two types of adsorption process,

1. **Physisorption** or physical adsorption: The process in whichadsorbateadsorbed on the surface of adsorbent through weakVan der Waals intermolecular forces.
2. **Chemisorption** or chemical adsorption is the adsorption in which a molecule adsorbed on surface through the formation of a chemical bond. In chemisorption molecule adheres on the surface more strongly than physisorption.

Adsorption of synthetic dyes on efficient and inexpensive solid adsorbent (activated carbon) has been considered a simpleand economical technique for removal of dye from wastewater, producing clear water which making it an effective alternative for the water treatment, especially when the adsorbent is less expensive. Adsorption is better than other techniques for wastewater treatment in terms of flexibility and simplicity of design, initial cost, ease of operation, sensitivity to toxic pollutants and nontoxic adsorbent after adsorption.

Activated carbon is most reliable and effective physicochemical treatment for removal of dyes. Activated carbons of commercial grade are very expensive. This produces the need to find effective activated carbons at low cost which can be applied to polluted water treatment. Many researches are going worldwide to find a low cost activated carbon for the dye polluted

wastewater treatment. A wide variety of low cost activated carbon such as bagasse pith, clay minerals, wood,neem saw dust, maize cob, orange peel wasused as viable replacement for commercial grade activated carbon for the removalof dyes from colored wastewater. The adsorption capacities of the low costactivated carbons are not high, thus the search for new adsorbents are still going on. Commercial grade activated carbons can be applied to different types of pollutants in wastewater. If low cost non-conventional precursors are used to prepare activated carbons for particular pollutants, then they can be economical for treatment of wastewater.

2.4 ADSORBATE

Congo red is the sodium salt of 3,3'-([1,1'-biphenyl]-4,4'-diyl)bis(4-aminonaphthalene-1-sulfonic acid)(formula: $C_{32}H_{22}N_6Na_2O_6S_2$; molecular weight: 696.66 g/mol). It is secondary diazo dye. Congo red is water-soluble, yielding a red colloidal solution.

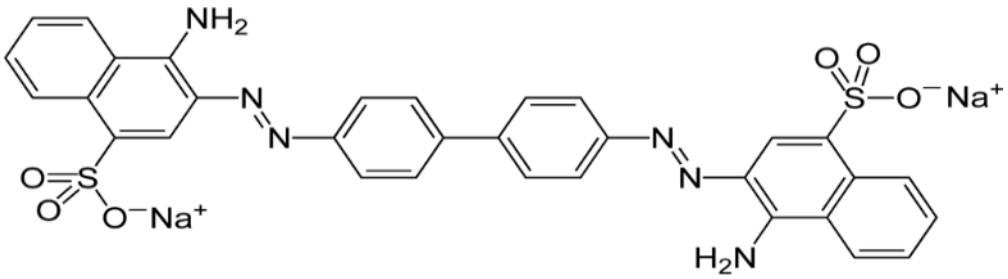


Fig 2-1: Congo red dye structure

Congo red was first synthesized in 1883 by Paul Bottiger. It has a strong, though apparently noncovalent, affinity to cellulose fibers. It is used in cotton textile, wood pulp and paper industries.It is toxic in nature and tendency to run and change colour when touced by sweaty fingers.

Congo red has a propensity to aggregate in aqueous and organic solutions. The proposed mechanisms suggest hydrophobic interactions between the aromatic rings of the dye molecules, leading to a pi–pi stacking phenomenon. Although these aggregates are present under various sizes and shapes, the "ribbon-like micelles" of a few molecules seem to be the predominant form (even if the "micelle" term is not totally appropriate here). This aggregation phenomenon is more

prevalent in high Congo red concentrations, at high salinity and/or low pH. It has uses in dyeing activities and diagnostic purposes.

Mall et al.,(2005) studied Removal of congo red from aqueous solution by bagasse fly ash and activated carbon. The effective pH was found to be about 7 and optimum contact time was found to be 4 hrs. Optimum BFA, ACC and ACL dosages were found to be 1, 20 and 2 g respectively.

Flavio et al.,(2008) studied Removal of Congo red from aqueous solution by anilinepropylsilica xerogel. Maximum adsorption capacity was found to be 22.62mg/g and the optimum contact time was found to be 20 min.

Vijaykumar et al.,(2009) studied Removal of Congo Red from Aqueous Solutions by Perlite. The adsorbent was characterized by Fourier Transform Infrared spectroscopy (FTIR), X-ray Diffraction (XRD) and Scanning Electron Microscopy (SEM). Adsorption experiments were carried out as batch studies at different adsorbent dose, pH, temperature and initial dye concentration. The dye adsorption equilibrium was rapidly attained after 40 min of contact time.

Abbas et al.,(2011) did a comparative study of removal of cogo red from aqueous solutions with raphanus sativus peels and activated carbon. The maximum adsorption capacity for radish was found to be 0.49 mg/g and for activated carbon 0.33 mg/g. It was also found that pH was the major governing factor in this case study.

Patel et al.,(2012) studied removal of congo red dye from its aqueous solution using natural coagulents. They found that the maximum percentage CR removal was found to be 98.0, 94.5 and 89.4 for SSP, Chitosan and MSP, respectively, at pH 4.0, coagulant dose of 25 mg/l, flocculation time 60 min and temperature of 340 K.

Chapter 3
Materials and Methods

3. MATERIALS AND METHODS

3.1 MATERIALS

Instruments used in this study with their manufacturers, functions and the operating conditions are listed below,

Table 0-1: List of instruments used in this study with details.

Instrument	Manufacture	Function	Operation conditions
Analytical balance	Sartorius (BS223S)	Weight measurement	100mg - 20g
pH meter	Systronics (361)	pH measurement	pH 1 to 14
Incubator shaker	REICO	Shaking of conical flasks	Speed: 100 rpm.
			Temperature: 20°C-40°C.
Scanning Electron microscopy	FEI Nova NanoSEM 230 FESEM	To study the surface structure and chemical composition	Magnification: up to 10000X
			Resolution : 1μm
UV-spectrophotometer	Labindia	To determine the absorbance	Wavelength-497nm
(BET) Surface Area Analyzer	Quantachrome Instruments	To determine the pore size and surface area of the sample.	Degassing of N_2 gas at 70^0C
Fourier Transform Infrared spectroscopy (FTIR)	Perkin-Elmer	To predict the organiccompounds present in thesamples	Resolution of 400 cm-1
			Range 400-4000 cm-1
			Temp-500 ^{O}C
Hot Air Oven	WEIBER	For drying of samples	Done at 60°C
Muffle Furnace	WEIBER, ADCO	For proximate analysis Preparing carbon	As per standards

3.1.1 Chemicals and glassware

All the reagents used during the adsorption study were of analytical grade and all the solutions were prepared using distilled-water. Glassware used for the experiments such as volumetric flasks, weighing cylinder, pipette etc. are procured fromBorosil Company andTarson Product Private Limited. All the glassware were rinsed thoroughly with tap water,subsequently with distilled water and dried in hot air oven to remove any trace of moisture present. Other chemicals used in the experiments were Potassium Iodide (Merck specialties Pvt. Ltd.), Iodine resublimed (Himedia Laboratories pvt.Ltd.), Sodium Thiosulphate (LobaChemiepvy.Ltd.), Orthophosphoric acid (Fisher Scientific).

3.2 METHODS

3.2.1 Preparation of adsorbent

Groundnut cake was obtained from the local market and was subjected to pretreatment to prepare the adsorbent. Initially the groundnut cake was dried to remove traces of oil in it, and then the cake was ground into powder form after which it was washed to remove any adhering impurities in it and then dried at 90 ^{0}C for 24 hours. Then proximate analysis of the sample was done to gauge its suitability for activated carbon preparation. The dried groundnut cake was then impregnated chemically using KOH solution and H_2SO_4 solution for a period of 5-6 hours. 3 samples each were prepared. Samples prepared with KOH were exposed to varying impregnation ratio such as0.5, 1 and 1.5 and for H_2SO_4, with 5%, 10%, and 15% acid concentration.The impregnated cake was then dried in the hot air oven for 24 to 48 hours at the temperature of 110^{0}C. The dried impregnated cake was then again grounded to fine particle size in the grinder. A sample of this groundnut cake was then pyrolysis at a temperature of 300^{0}C in a Tubular furnace using N_2 gas at a flow rate 10 cm^3/min for 2 hours. After pyrolysis the activated carbon obtained from KOH was again grounded to the fine powder and then washed several times first with 0.1 N HCL till no change in pH was observed. Similarly H_2SO_4 impregnated samples was washed with 0.1N NaOH solution and then with distilled water to remove ash content from the carbon. It was subsequently dried in the hot air oven at 45^{0}C.

3.2.2 Batch Experimental Procedure:

Adsorption studies were conducted using50 mg/l CR dye solution was prepared.First the calibration curve was plotted to obtain dye concentration in the sample using UV spectrophotometer at wavelength of 479nm. Then the effect of contact time, adsorbent dosage, initial dye concentration, solution pH and temperature were measured by varying the different parameters, keeping in shaker and then measuring the dye concentration in the final solution using UV spectrophotometer and calibration curve.

3.3 METHODS FOR CHARACTERIZATION OF SAMPLE

3.3.1 Proximate analysis

The proximate analysis of a substance determines distribution of products when a sample is subjected to high temperature under specified conditions. Proximate analysis separates the products into its four contents: (1) moisture, (2) fixed carbon, (3) volatile matter and (4) ash. It is the most often analysis usedfor characterizing a material.

Volatile matter:

Volatile content of the sample indicate the combustible matter of the sample when subjected to high temperature. 1g sample was weighed and takenin a closed lid crucible. The crucible was heated to 925 ^{0}C for 7.5 min in a muffle furnace. After heating the crucible was taken out and cooled in a desicator and then weighed,

$$\mathrm{Vm} = \frac{(\mathrm{W2} - \mathrm{W3})}{(\mathrm{W3} - \mathrm{W1})} * 100 \qquad \text{……. (3.1)}$$

Ash content:

Presence of ash indicates the density and combustible material present in the sample. 1g sample was weighed and taken into anopen crucible. Cruciblewith sample was heated to 750 ^{0}C for 1.5 hr. After heating, crucible was taken out and cooled in a desicator and then weighed.

$$\mathrm{A} = \frac{(\mathrm{W3} - \mathrm{W1})}{(\mathrm{W2} - \mathrm{W1})} * 100 \qquad \text{……. (3.2)}$$

Moisture content:

Moisture content indicates the quantity of water contained in a sample. 1g of sample was weighed and taken in a petri dish. Sample was spread uniformly on the petri dish. Petri dish was then heated at 105 ^{0}C for 1.5 hr in a hot air oven. After heating the petridish was taken out and cooled in desicator and then weighed,

$$M = \frac{(W2 - W3)}{(W2 - W1)} * 100 \qquad \text{……. (3.3)}$$

Fixed carbon:

Fixed carbon indicates solid combustible residue that remains after volatile matter is removed. The fixed-carbon content of samplewas determined from moisture, volatile matter, and ash contentsof the sample.

$$\% \text{ Fixed carbon} = 100 - (V_m + A + M) \qquad \text{……. (3.4)}$$

3.3.2 Thermo-Gravimetric Analysis

Pyrolysis is the heating of a substance in the absence of air at a specifictemperature. To understand the change in mass of the groundnut cake with temperature and time, thermo-gravimetric analysis was performed. In Thermo gravimetric analysis (TGA)weight of sample is measured as a function of temperature or time whensample was heated in a controlled atmosphere. A little amount of groundnut cake was taken and heated up to a final temperature of 800 ^{0}C. TGA was performed in controlled atmospheres at a heating rate of 25 ^{0}C/Min. Weight lose curve was plotted against temperature which provides the temperature range in which thermal degradation of groundnut cake was takes place.

3.3.3 Yield

The raw material was taken in a crucible and weighed. Then it was pyrolysed and cooled in a dessicator and weighed again. Yield is given as

$$[(W_2\text{-}W_3)/W_2 - W_1] * 100$$

3.3.4 Iodine value test

The adsorption of aqueous I_2 is gives an indication aboutadsorption sits present in thesample. The iodine value, the amount of aqueous iodine adsorbed per gram of activated carbon at equilibrium with 0.1N Iodine solution, was measured according to the procedure set by the American Society for Testing and Materials (ASTM 2006). Iodine Number is the most acceptable fundamental test used to characterize activated carbon. Iodine value gives the measure of unsaturation level (higher Iodine number indicates more adsorption sits present on the activated carbon) of the sample.

- 0.1N Iodine solution
- 0.05N Sodium Thiosulphate solution
- 1% Starch solution
- Activated carbon

Procedure of the iodine value test:

1. *Standardization of Iodine solution,*

- 10ml of 0.1N Iodine solution was taken in conical flask.
- 2 drops of Starch indicator was added to the flask.
- Pale yellow color of Iodine Solution turned Blue.
- The solution was titrated with 0.05N Sodium Thiosulphate solution till it becomes Colorless.
- Burette reading was noted as blank reading (B).

2. *To test iodine value of activated carbon,*

- 0.2 g of Activated carbon was weighed.
- Weighed activated carbon was taken into completely dry conical flask.
- 40ml of 0.1N Iodine solution was then added the flask.
- The flask was shaken for 4 minutes and then solution was filtered.
- The filtrate collected in a completely dry flask and then 10ml of the filtrate was titrated with0.05N Sodium thiosulphate solution using starch as indicator until colorless solution appear.
- Burette reading noted asactivated carbon reading (A).

Now Iodine value can be calculated using the equation below,

$$\text{Iodine value} = C \times \text{Conversion factor (mg/g)}$$

Where,

$$\text{Conversion factor} = \frac{(253.81) * \text{normality of iodine solution} * 40}{\text{Wt. of carbon} * B}$$

$$C = B - A$$

3.3.5 SEM analysis

Scanning electron Microscopy (SEM) provides information about the surface morphology of sample. In SEM emitted electrons from an electron gun strike the surface of sample and generateother low energy secondary electrons. Intensity ofthe generated secondary electrons from surface of the sample waschanged by the surface topography of the sample. An image based on the intensity of secondary electron was constructed as a function of the arrangement of the scanning primary electron beam. The preparation of the sample is done by coating it with gold to avoid ionization of the sample.

3.3.6 FTIR analysis

FT-IR stands for Fourier Transform Infrared, used to know the chemical structure of the sample. In FTIR spectroscopy, Infrared radiation is passed through the sample, from which some of the radiation is absorbed within the sample and some radiation is passed transmitted by the sample. The resulting spectrum from absorbance and transmitting of infrared by sample was represents the molecule absorption and transmission, which creating a molecular identification image of the sample. Every molecule and molecular bonds formed a different infrared spectrum at the radiation receiver. Therefore, FTIR spectroscopy give result toidentify the (qualitative analysis) of different kind of bonds and structure of the sample. In the present study FTIR of groundnut cake, activated carbon before adsorption and activated carbon after adsorption was done.

3.4 ADSORPTION STUDY

3.4.1 Study the effect of contact time:

To study the influence of contact time on the adsorption of CR dye, 100 ml of 50mg/l dye solution taken in a conical flask and 1g of activated carbon was added in the flask at solution

pH.The flask was kept at 37^0C in the shaker at 100rpm shaking speed. Then the sample was pipetted out at the interval of 20 min for 160 minutes. The dye concentration in the remaining sample was analyzed for absorbance in the UV-spectrophotometer. And the time after which the change in adsorption capacity became negligible was taken as the equilibrium contact time.

3.4.2 Study the effect of initial solution pH:

One of the important factors that affect the adsorbent capacity in wastewater treatment is pH of solution. To study the effect of pH on the adsorption of CR dye, 100 ml of 50mg/l dye solution taken in a conical flask and then different pH of the solution (2 to 8) were maintained in the different conical flasks, and 1g of activated carbon was added in every flask. Next the conical flask was kept at 37^0C in the shaker at 100rpm. The sample was taken out after the equilibrium contact time and then the remaining dye concentration in the flask was calculated spectrophotometrically. The pH of the solution for which the adsorption capacity is maximum is taken as the optimum pH of the solution.

3.4.3 Study the effect of adsorbent Dosage:

Effect of adsorbent Dosage on the adsorption of CR dye was studied by taking 100 ml of 50mg/l dye solution in a conical flask and then different amount of activated carbon (0.5 to 2.5 g) were added in the different conical flask at 6 pH. After we keep the conical flask at 37^0C in the shaker at 100rpm, the samples were taken out after 120 min to obtain the concentration of the remaining dye in the solution after adsorption.

3.4.4 Study the effect of temperature:

To study of the effect of temperature on the adsorption is done, 75mg/l of the dye solution in the conical flask and 1.5g of activated carbon was added to it and the after which the flask was kept in the shaker at different temperature(20, 30 40 ^{0}C), and at 100 rpm and at 6pH.

In all the study of different parameters, the absorbance capacity (q_e) of the activated carbon was calculated by the equation,

$$qe = \frac{(Co - Ce) * V}{W} \qquad \text{……. (3.5)}$$

and % removal was calculated by the equation,

$$q\,\% = \frac{(Co - Ce)}{Co} * 100 \qquad \text{……. (3.6)}$$

3.5 ADSORPTION KINETIC STUDIES

Adsorption kinetics study is done for the understanding of the rate of adsorption, mechanism of adsorption, which are the two most important factors for the optimal design for the practical application. To analyse the adsorption kinetics of CR over activated carbon four models; Pseudo-1st, Pseudo-2nd order, Elovich equation, and intra-particle diffusion were tested.

3.5.1 Pseudo-1st order equation:

In this equation it is assumed that the rate (dq_e/d_t) is proportional to the difference between the amount of adsorption at time t and the adsorption capacity of adsorbent ($q_e - q_t$). Let K_1 be the proportionality constant, then,

$$\frac{dq}{dt} = K1(qe - q) \qquad \text{……. (3.7)}$$

Linear form of above equation can be written as,

$$\ln\frac{(qe - q)}{qe} = -K1 * t \qquad \text{……. (3.8)}$$

3.5.2 Pseudo-2nd order equation:

Pseudo-second order kinetics based on the equilibrium adsorption capacity is expressed as

$$\frac{dq}{dt} = K2(qe - q)^{2} \qquad \text{……. (3.9)}$$

Linear form of the above equation can be get by integrating the equation,

$$\frac{t}{qt} = \frac{1}{K2 * qe2} + \frac{t}{qe} \qquad \text{……. (3.10)}$$

The value of q_e and K_2 can be estimated from slope and intercept respectively from linear plot between t/q_t versus t.

3.5.3 Elovich equation:

The Elovich equation used for general application to chemisorption with a wide range of slow adsorption rate,

……. (3.11)

$$\frac{dq}{dt} = \alpha * e\text{^}(-\beta\alpha)$$

The linear form of the above equation is,

$$q = \frac{\ln(\alpha * \beta)}{\beta} + \frac{\ln t}{\beta} \qquad \text{……. (3.12)}$$

Where α and β can be determine by intercept and slope of the plot between q versus ln(t).

3.5.4 Intra-particle diffusion model:

The intra-particle diffusion model is based on the assumption that the adsorption uptake q_t varies proportionally with square root of contact time,

$$qt = Kid * \sqrt{t} + I \qquad \text{……. (3.13)}$$

Where I is the intercept (mg/g) and k_{id} (mg/g.$min^{1/2}$) is inter particle diffusion rate constant and can be obtained from slope of the plot between q_t versus √t.

3.6 ADSORPTION EQUILIBRIUM STUDY

Adsorption is usually described by the isotherm, which give a relation between adsorbed amount of adsorbate and the concentration in bulk phase at constant temperature at equilibrium. Here Langmuir, Freundlich, models were applied to fit the equilibrium data.

3.6.1 Langmuir adsorption isotherm:

The Langmuir isotherm is based on the assumption that there was only one layer of molecules adsorbed on to the surface, i. e. monolayer adsorption of the adsorbate. The monolayer isotherm is represent by the equation,

$$qe = \frac{qm * b * Ce}{1 + b * Ce} \qquad \text{…. (3.14)}$$

On integrating the above equation we get the linear form of the above equation as,

$$\frac{Ce}{qe} = \frac{Ce}{qm} + \frac{1}{qm * b} \quad \text{……(3.15)}$$

The value of b and q_m can be obtained from the plot between C_e/q_e and C_e.

3.6.2 Freundlich isotherm:

Freundlich adsorption isotherm is based on the assumption that the distribution of the heat on the adsorbent surface is non-uniform, namely a hertogeneous adsorption.

Freundlich model can be express as,

$$qe = Kf * Ce\hat{}(\frac{1}{n}) \quad \text{……. (3.16)}$$

Linear form of the above equation is written as,

$$\ln qe = \ln Kf + 1/n \ln Ce \quad \text{……. (3.17)}$$

The value of K_f and n can be determined from the plot between $\ln q_e$ versus $\ln C_e$.

Chapter 4
Results and Discussion

4. RESULTS AND DISCUSSION

4.1 CHARACTERISATION OF GROUNDNUT CAKE

Characterization of precursors is essential to know the ash and volatile content of the precursor. It indicates the available carbon in the form of fixed carbon to obtain the activated carbon.

4.1.1 Proximateanalysis

Proximate analysis of the groundnut cake gives the following results,

Table 0-1: Proximate analysis of groundnut cake

Content	Percentage (%)
Moisture content	12.23
Volatile matter	75.72
Ash content	4.81
Fixed Carbon	7.24

The above table indicates that the groundnut cake has high volatile and moisture content which leads to very low fixed carbon as compared to commercial activated carbon which was prepared from coconut shell. This may be due to the fact that coconut shell is a harder material and groundnut cake is an extracted residue.

4.2 Optimization of process parameters for preparation of activated carbon from groundnut cake

4.2.1 Thermogravimetric analysis

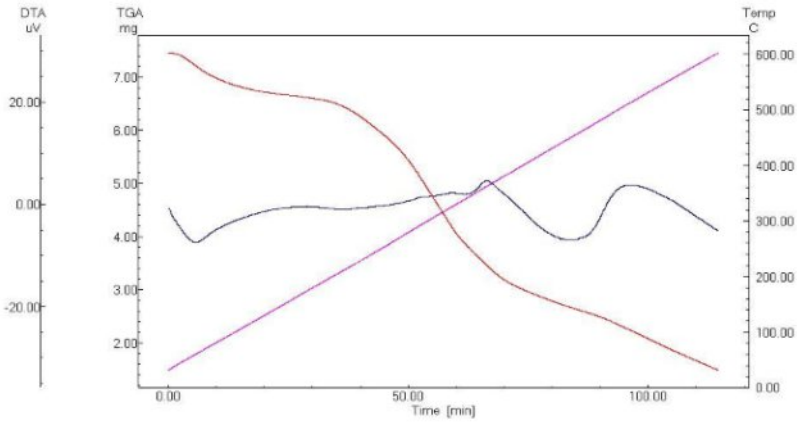


Fig 4-1: TGA analysis of Groundnut cake

From the Thermo gravimetric analysis of the groundnut cake sample, it can be seen that the optimum temperature for pyrolysis of sample for preparation of activated carbon is 300^{o}C.

4.2.2 Yield

Activated carbon was prepared using different concentrations of acidic and basic activating agents and their yield and iodine no. were measured to determine optimum conditions for preparation of activated carbon from groundnut cake.

Table 4-2: Yield of activated carbon

Potassium hydroxide Impregnation ratio	**Yield(%)**	**Sulfuric acid conc. (vol./vol.)(%)**	**Yield(%)**
0.5	64.43	5	68.98
1	64.71	10	71.69
1.5	67.48	15	70.28

4.2.3 Iodine value test

Table 4-3: Iodine number of activated carbon

Potassium hydroxide Impregnation ratio	**Iodine number**	**Sulfuric acid conc. (vol./vol.)(%)**	**Yield(%)**
0.5	325	5	550
1	535	10	696
1.5	495	15	603

The yield (71.69%)as well as iodine number(696) of the activated carbon sample prepared from 10% sulfuric acid was found to be maximum which indicates that sulfuric acid is a better activating agent in this case than potassium hydroxide. Therefore, activated carbon prepared from groundnut cake with 10% sulfuric acid as the activating agent was used for future work.

4.3 CHARACTERISATION OF THE ACTIVATED CARBON

4.3.1 Proximate analysis

Proximate analysis of the groundnut cake activated carbon gives the following results,

Table 0-4: Proximate analysis of groundnut cake activated carbon

Content	**Percentage (%)**
Moisture content	12.68
Volatile matter	15.43

Ash content	9.57
Fixed Carbon	62.32

4.3.2 Yield

The yield of the sample was found to be **71.69%.**

4.3.3 Iodine value test

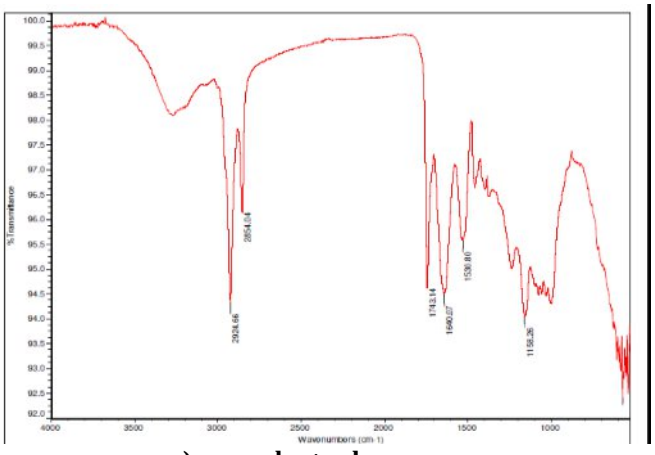
Iodine value test give the information about adsorption sites available at the adsorbent for the adsorption. The higher the Iodine number the higher the adsorption capacity of the activated carbon for adsorption.

In the present work the iodine number of the groundnut cake activated carbon obtained was **696.**

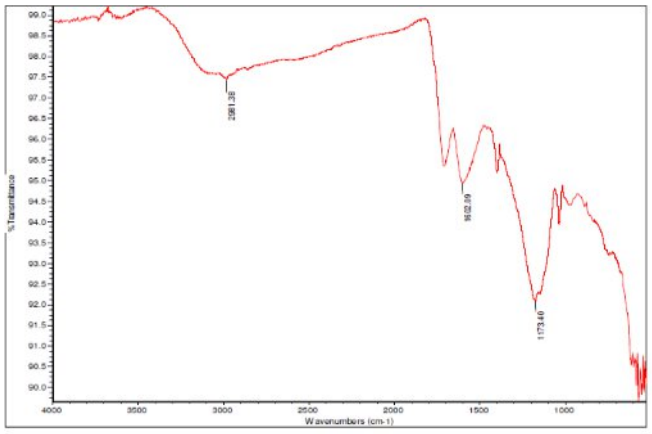
4.3.5 FTIR analysis

FTIR spectra of groundnut cake, activated carbon before and after adsorption are presented in figure 4-2. The peaks around 2800 and 2900 cm^{-1} attribute to C-H stretches (alkanes)in the groundnut cake. The spectrum around 1500 cm^{-1} shows the conversion of C-H to C=C (alkenes) which confirms the activation of the groundnut cake. The molecular group found in the activated carbon after adsorption consist spectrum band 1550-1700 cm^{-1} which shows the molecular group of amines(NH_2) and nitriles(C=N). The presence of theses groups in the activated carbon after adsorption IR spectra indicates the adsorption of the CR dye on the activated carbon.

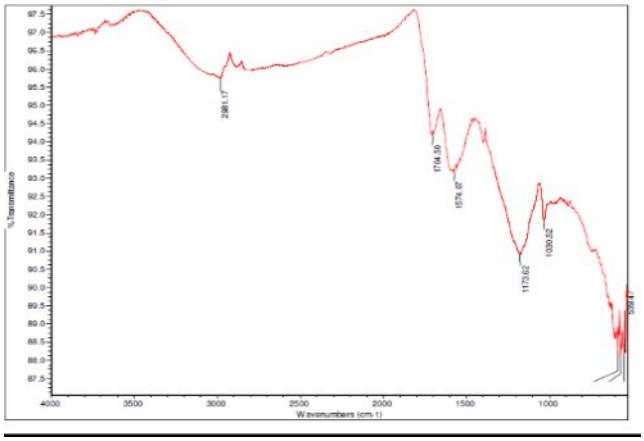
Fig 4-2: FTIR images:



a) groundnut cake



b) activated carbon before adsorption



c) activated carbon after adsorption

4.3.6 SEM analysis

SEM is the tool for surface characterization of the sample.Fig. shows images of activated carbon before and after adsorption. It is seen that there is a good chances of adsorption of the CR dye on the activated carbon as there are many pores available on the surface of the activated carbon (fig.4-3(a)) while fig.4-3(b) shows the activated carbon after adsorption, in which it was clearly seen that there are a layer of the CR dye and pores on the surface of groundnut cake activated carbon was filled. The dye molecules seems to form a void free film on the surface of the activated carbon which shows a good adsorption of the dye on the adsorbent .

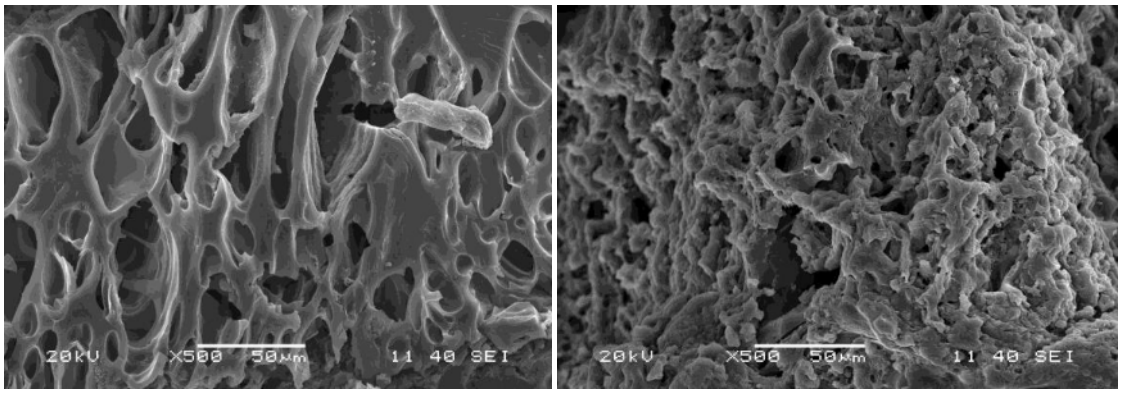


Fig 4-3: SEM images:

a)before adsorption **b) after adsorption**

4.4 ADSORPTION STUDIES

Adsorption studies include the study of effect of contact time, solution pH, adsorbent dosage, initial dye concentration and temperature on adsorption capacity.

4.4.1 Effect of contact time

Study of effect of contact time on adsorption of dye on activated carbon is necessary to find out the optimum contact time for adsorption capacity to reach equilibrium and thus find our the optimum time required for effective adsorption. Effect of contact time was studied at temp. 37^{o}C, solution pH, 100rpm speed, adsorbent dosage of 1g/100 ml and initial dye concentration of 50 mg/l. From Fig. 4-4 it can be seen that initially the adsorption capacity increases with time upto 120 mins after which the change in adsorption capacity becomes negligible. Thus 120 mins is taken as the optimum contact time for adsorption of CR dye on groundnut cake activated carbon.

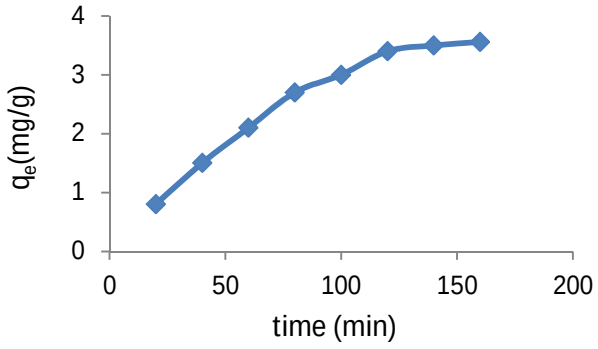


Fig 4-4: Effect of contact time on adsorption of CR dye on activated carbon

4.4.2 Effect of initial solution pH

The pH of the dye solution has significant effect on the dye adsorption. Solution pH alters the charge on surface of adsorbent as well as the extent of ionization of aqueous adsorbate species in the solution and consequently the rate of adsorption. The change in the pH affected the adsorption capacity due to the presence of functional group at the adsorbent and adsorbate surface. Effect of pH was studied at 37^{o}C temp., 100 rpm speed, adsorbent dosage of 1g/100 ml and initial dye conc. Of 50 mg/l. It was observed from the fig. 4-5 that as we increased the pH of the dye solution from 2 to 10 the adsorbent capacity of the activated carbon was affected significantly. As we increased the pH from 2 to 6 the adsorption capacity increased from 1.5 to 2.6 mg/g, but again as we further increased the pH of the solution from 6 to 10 it was found that the adsorption capacity reduced from 2.6 to 2.3 respectively. This was attributed to the fact that as the pH of the solution increase the negatively charge ion of the solution neutralized the positive charge of the surface of the activated carbon that caused the reduction of the adsorbent capacity of the activated carbon .

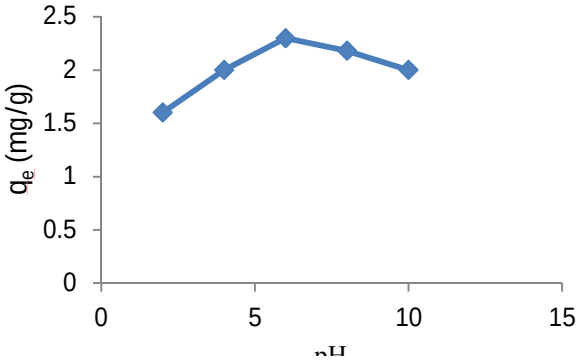


Fig 4-5: Effect of pH of dye solution on adsorption capacity of congo red dye

4.4.3 Effect of adsorbent dose

Use of optimum dose of the activated carbon in the adsorption is a crucial factor for the cost effective industrial application of the process. Effect of adsorbent dosage was studied at 37^{o}C temp., pH 6, 100 rpm speed and initial dye conc. Of 50 mg/l. It can be seen from the fig. 4-6 that as we increase the adsorbent doses from 0.25 g/l to 1.5 g/l the adsorption capacity increased from 1.8 to 2.6 mg/g but on further increasing the adsorbent dose from 1.5 to 2.5 g/l, the adsorption capacity decreased from 2.6 to 1.9. The increase may be attributed to the increase in the adsorption sites as the amount of activated carbon increases. The decrease may be due to the saturation of active sites with increase of activation sites.

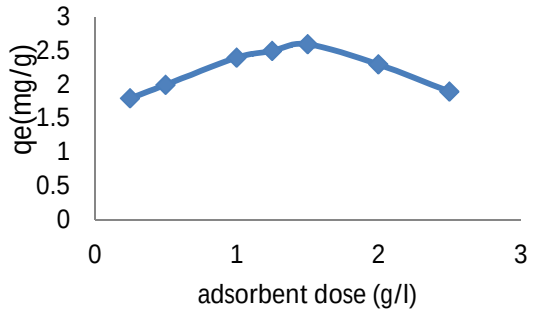


Fig4-6: Effect of adsorbent dosage on adsorption capacity of congo red dye

4.4.4 Effect of initial dye concentration

Effect of initial dye conc. was studied at 37^{o}C temp., pH 6, 100 rpm speed and adsorbent dosage of 1.5g/100 ml.It can be seen that as the initial dye conc. Is increased from 25 to 75 mg/l, the adsorption capacity of the groundnut cake increased from 1.2 to 4 mg/g but on further increasing the dye concentration from 75 to 100 mg/l, the adsorption capacity decreases to 3.9 mg/g. The increases can be attributed to the fact that more dye will be adsorbed on the adsorption sites as the dye concentration is increased. As the initial dye conc. Increases, conc. Gradient inc. which inc. the driving force to reach equilibrium. The initial rate of adsorption of CR dye on the activated carbon is higher for higher dye conc. Due to low resistance to dye uptake on the adsorption sites. The subsequent decrease may be due to saturation of active sites on the activated carbon.

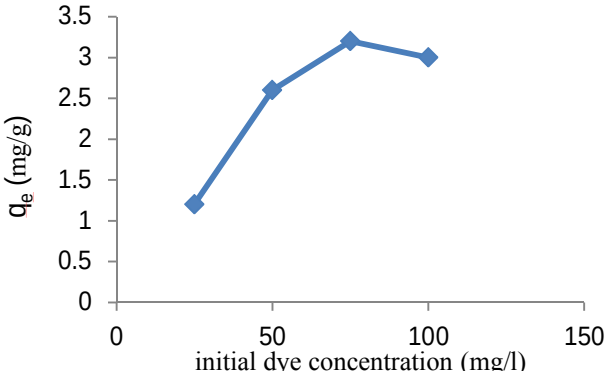


Fig4-7: Effect of initial dye concentration on adsorption capacity of congo red dye

4.4.5 Effect of the temperature

The effect of temperature was studied at the temperature 20, 30,40^{0}C, at the 6 pH, with 75 mg/l of dye concentration and at 100 rpm and with activated carbon dose of 1.5 g /100 ml of dye

solution. Results indicate that the adsorption capacity of activated carbon for congo red dye increased with temperature. This may be a result of increase in the mobility of the latge dye ion with temperature. An increasing no. of molecules may also acquire sufficient energy to undergo an interaction with active sites at the surface. Furthermore, increasing temperature may produce a swelling effect within the internal structure of the activated carbon enabling large dyes to penetrate further.

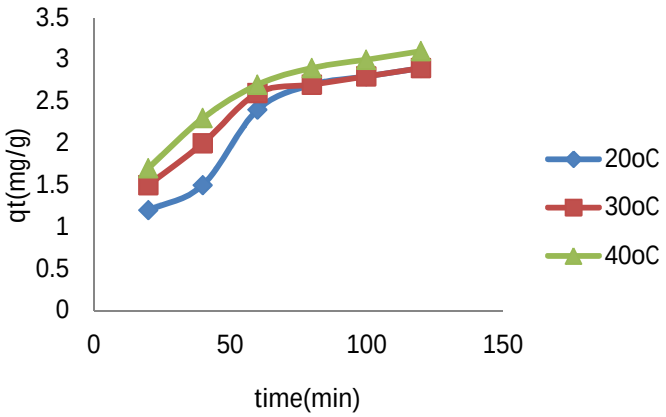


Fig4-8: Effect of temperature on adsorption capacity of congo red dye

4.5 ADSORPTION KINETIC STUDIES

In the present study kinetics of the adsorption of MG on the activated carbon was tested at three different temperatures, as 20, 30 and 40 ^{0}C. Following table shows the parameter of the four different kinetics models.

Table 0-6: Parameters of different kinetic models at different temperatures

Kinetic model	**Constants**	**Value (**at different temperatures**)**		
		20 ^{0}C	30 ^{0}C	40 ^{0}C
Pseudo -1st order	K_1	0.040	0.036	0.034
	R^2	0.966	0.983	0.953
Pseudo- 2nd order	q_e	4.524	3.610	3.731
	K_2	0.0036	0.0098	0.011
	R^2	0.926	0.993	0.999
Elovich modal	A	0.16	0.292	0.402
	B	1.002	1.297	1.319
	R^2	0.926	0.966	0.991

Intra-particle diffusion	K_{id}	0.302	0.229	0.223
	I	-0.167	0.583	0.825
	R^2	0.936	0.932	0.977

From the above table, the experimental data shows that the adsorption of CR can be described better by the Pseudo-2nd order kinetic model in term of higher correlation coefficient $R^2 > 0.9995$.

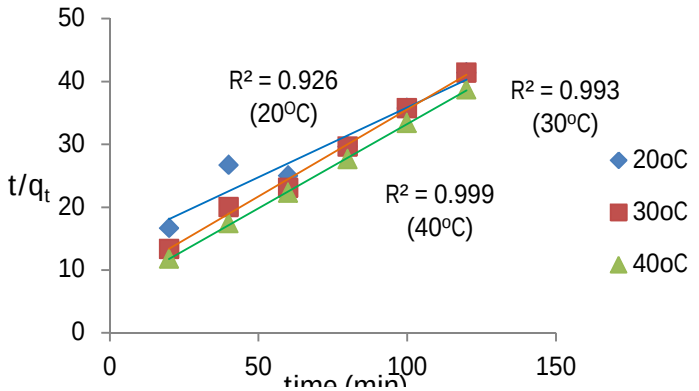


Fig-4-9: Pseudo 2nd order kinetic plots for adsorption of congo red dye at different temperatures

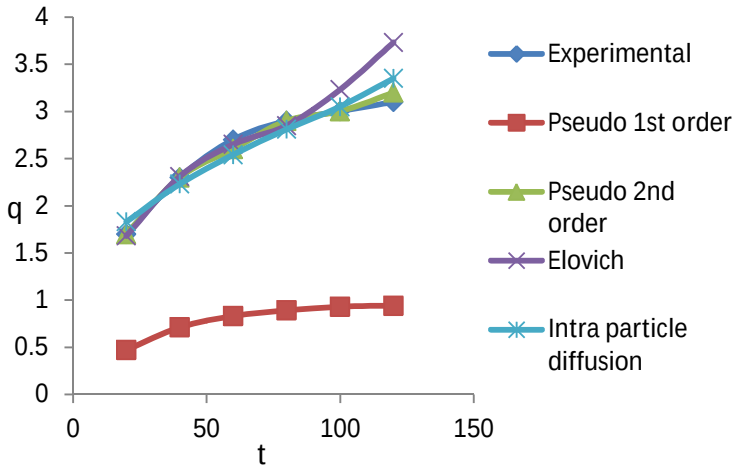


Fig-4-10: Adsorption rate curves at 40 ^{0}C

From the figure, it can be seen that Pseodo 2nd order kinetic model best fits the experimental data.

4.6 ADSORPTION EQUILIBRIUM STUDIES

The correlation coefficient and other parameter of the Langmuir and Freundlich isotherm are listed below.

Table 0-7: Adsorption isotherm parameter at different temperatures

Isotherm models	**Constants**	**Value** (at different temperatures)		
		20 ^{0}C	30 ^{0}C	40 ^{0}C
Langmuir	q_m	41.7	38.5	71.4
	b	0.001	0.002	0.194
	R^2	0.782	0.882	0.479
Freundlich	K_f	0.073	0.104	0.091
	1/n	0.909	0.846	0.904
	R^2	0.965	0.992	0.992

From the data listed in the table 4-5, it was observed that Freundlich adsorption model was found to fit the experimental data with high correlation coefficient (0.992) as compared to the Langmuir adsorption isotherm (0.882) at the same temperature.

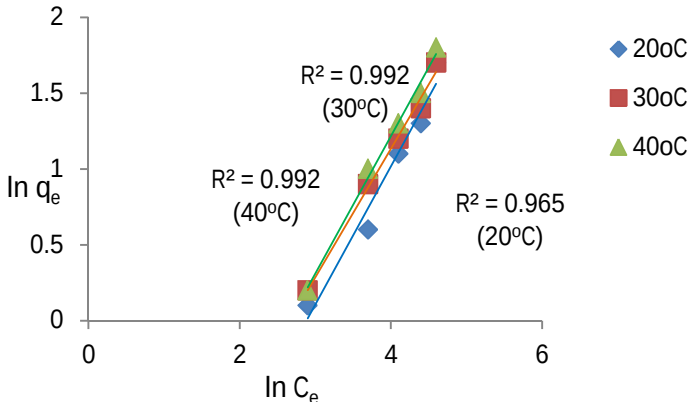


Fig4-11: Freundlich isotherm plots for adsorption of congo red dye at different temperatures

From fig. 4-11, it is observed that Freundlich isotherm gives theoretically linear plots with high correlation coefficient (R^2) which shows the applicability of Freundlich isotherm for the adsorption of CR on the groundnut cake activated carbon under all three temperatures. Hence Freundlich isotherm better described the adsorption behavior at equilibrium than Langmuir isotherm.

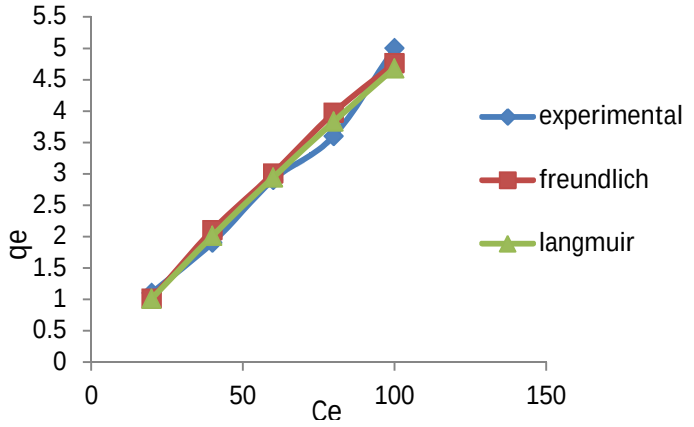


Figure 0-12: Adsorption isotherms at 40 ^{0}C

From fig. 4-12, it is observe that from the two models (Freundlich and Langmuir) it the Freundlich adsorption isotherm is most adopted fitting adsorption isotherm and Freundlich isotherm gives the maximum adsorption capacity at the equilibrium.

Chapter 5
Conclusions

5. CONCLUSIONS

Groundnut cake was successfully used as a cheap precursor for removal of Congo Red dye from aqueous solution. Following conclusions are made by the present study of removal of CR dye by groundnut cake activated carbon from aqueous solution.

- The optimum conditions preparation of activated carbon was found to be 300^{o}C pyrolysis temperature and 10% sulfuric acid as the activating agent.
- The shifting of peaks in FTIR spectrum and images from SEM confirms the adsorption of CR dye on groundnut cake activated carbon.
- Groundnut cake activated carbon has surface are of 34 m^2/g, 15.4 % fixed carbon and Iodine no. as 696.
- The optimum conditions for adsorption was found to be 75 mg/l (initial dye concentration),1.5 g/100ml (activated carbon dose), 6pH, 40 ^{0}C and 100 rpm shaking speed.
- At these optimum conditions experimental values were validated with Frendulich isotherm models which were fitted well to the experimental data (R^2 = 0.992).
- The kinetics of the adsorption process agreed well to the Pseudo-2nd order model.

Thus the present study shows that groundnut cake activated carbon can be used as an inexpensive and efficient adsorbent for removal of CR dye from aqueous solution.

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